NASA-CR-121123
043680-19-T

N73 15919

THE UNIVERSITY OF MICHIGAN

COLLEGE OF ENGINEERING
DEPARTMENT OF MATERIALS AND METALLURGICAL ENGINEERING

Progress Report

Time-Dependent Edge-Notch Sensitivity of Oxide and Gamma Prime Dispersion Strengthened Sheet Materials at 1000° to 1800°F (538° - 982°C)

DAVID J. WILSON
ALBERTO FERRARI

Supported by:

National Aeronautics and Space Administration
Grant NGL-23-005-005
Washington, D. C.

Administered through:

September 1972

OFFICE OF RESEARCH ADMINISTRATION • ANN ARBOR

T H E U N I V E R S I T Y O F M I C H I G A N

COLLEGE OF ENGINEERING
Department of Materials and Metallurgical Engineering

Progress Report

TIME-DEPENDENT EDGE-NOTCH SENSITIVITY OF OXIDE AND GAMMA PRIME DISPERSION STRENGTHENED SHEET MATERIALS AT 1000° TO 1800°F (538°-982°C)

David J. Wilson
Alberto Ferrari

ORA Project 043680

supported by:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GRANT NO. NGL-23-005-005
WASHINGTON, D.C.

administered through:

OFFICE OF RESEARCH ADMINISTRATION ANN ARBOR

September 1972

TABLE OF CONTENTS

LIST OF TABLES

Table

LIST OF FIGURES

Figure

LIST OF FIGURES (Concluded)

Figure

INTRODUCTION

Notch sensitivity of heat resistant sheet materials is being studied at The University of Michigan, Ann Arbor, Michigan. The research is sponsored by The National Aeronautics and Space Administration, Washington, D.C.

Severe time-dependent edge-notch sensitivity (Figure 1) of Waspaloy and Inconel 718 has been shown to occur when notched specimens were loaded below the approximate 0.2 percent smooth specimen offset yield strength and when test data from smooth specimens indicated that small amounts of creep strain consumed large fractions of creep-rupture life [1,2]. The susceptibility to notch sensitivity has also been correlated with dislocation motion mechanisms [2,3]. Coherent precipitates smaller that a "critical size" were sheared by dislocations. This gave rise to localized deformation and time-dependent notch sensitive behavior. Larger particles were by-passed by dislocations and the deformation was homogeneous. Under these conditions, no time-dependent notch sensitivity was observed.

Research is continuing to determine the generality, or scope, of the concepts that have been developed. Presently reported are results for two alloys:

(1) TD-Ni,Cr, which is strengthened by an incoherent dispersion of ThO_2 particles. This material was selected for study, because, dislocations would have to by-pass the oxide particles during creep. Therefore, the alloy was not expected to be susceptible to time-dependent notch sensitivity.

(2) An experimental nickel-base alloy, "Modified Waspaloy" having a relatively low volume fraction (0.095) of coherently precipitated γ', $Ni_3(Al,Ti)$. This material was studied in order to permit extension of the results for Waspaloy (γ' volume fraction 0.23) to alloys with less precipitate.

The TD-Ni,Cr was evaluated in the stress-relieved condition while the Modified Waspaloy was studied after heat treatments that produced a range of γ' particle sizes. Tensile and creep-rupture tests were carried out at 1000°-1800°F (538°-982°C) for TD-Ni,Cr and at 1000°-1400°F (538°-760°C) for Modified

Waspaloy. The microstructural features, particularly the dislocation structures in the tested specimens, were evaluated.

EXPERIMENTAL DETAILS

The commercially produced TD-Ni,Cr used in the investigation had the following reported composition (weight percent):

Ni	Cr	ThO_2	C	Si
78.72	19.24	2.01	0.03	0.003

The material was received as 0.015 in. (0.38 mm) thick, stress relieved, sheet.

The "Modified Waspaloy" was prepared as a vacuum induction melted ingot with aim composition similar to Waspaloy [1] except for lower amounts of titanium and aluminum (the Ti/Al ratios were essentially the same). The compositions were as follows (weight percent):

	Modified Waspaloy	Waspaloy		Modified Waspaloy	Waspaloy
Ni	Bal	Bal	Zr	0.031	0.03
Cr	19.40	19.33	B	NA*	0.005
Co	14.70	13.52	Fe	NA	0.55
Mo	4.10	4.16	S	NA	0.007
Ti	1.78	2.95	Mn	NA	<0.01
Al	0.67	1.35	Si	NA	0.05
C	0.053	0.06	Cu	NA	0.03

*NA = Not Analyzed

The ingot was radiographed to assure soundness, preheated at 2000°F (1093°C) and hot rolled in small reductions to a thickness of about 0.032 in. (0.81 mm) using reheats between passes. These sheets were pickled and cold reduced (20-25 percent) in several passes to a final thickness of approximately 0.025 in. (0.64 mm). Specimen blanks were individually solution treated 1/2 hr at 1975°F (1080°C) in an argon atmosphere and air cooled. To prevent warping, the blanks were aged in batches of 10 or 12 while clamped in a fixture. The aging treatments were: (a) 3 hr at 1325°F (718°C), (b) 1 hr at 1400°F (760°C), and (c) 16 hr at 1400°F (760°C).

For both alloys, smooth and edge-notched (K_t>20) specimens were machined in the longitudinal (rolling) direction. The specimen dimensions and test procedures were the same as previously reported [1]. For optical metallography, samples of Modified Waspaloy and TD-Ni,Cr were etched electrolytically in "G" etch [4] and a solution of 10 percent oxalic acid in water, respectively. The volume fraction of γ' in Modified Waspaloy was determined by electrollytic extraction [5] using duplicate samples solution treated 1/2 hr at 1975°F (1080°C) and exposed 1000 hr at 1400°F (760°C). Techniques for the preparation of thin foils for study by transmission electron microscopy were reported previously [3].

MECHANICAL CHARACTERISTICS

The results of tensile and creep-rupture tests are included as Table 1 for TD-Ni,Cr and Table 2 for Modified Waspaloy. The principal features, particularly those related to notch sensitive behavior, are presented in the following sections.

Rupture Strengths

For TD-Ni,Cr, the notched specimen rupture curve at 1000°F (538°C) was somewhat below that for smooth specimens (Figure 2). At higher temperatures, the rupture strengths for notched specimens were similar or higher than for smooth specimens. At all test temperatures, the notched to smooth rupture strength ratios (N/S) were greater than determined by tensile tests (Table 1), i.e., the alloy did not exhibit time-dependent notch sensitivity.

The heat treatment variations of Modified Waspaloy resulted in a range of smooth specimen strengths at the lower test temperatures. The strength increased as the aging treatment was changed from 3 hr at 1325°F (718°C) to 1 hr at 1400°F (760°C) to 16 hr at 1400°F (760°C). At the higher temperature and longer test times, the strengths were similar. (This resulted, at least in part, from γ' growth during the test exposures.) For all heat treated materials the N/S rupture strength ratios decreased with increasing time and/or temperature (Figure 3), i.e., time-dependent notch sensitivity was not observed.

Creep Resistance

The minimum creep rates determined from the smooth specimen tests are included in Tables 1 and 2. The most striking feature was that the stress-minimum creep rate curve (Figure 4) for TD-Ni,Cr at 1000°F (538°C) was considerably steeper than the curves at temperatures from 1200° to 1800°F (649°-982°C). This indicates that different mechanisms control creep in these two ranges of test conditions.

For Modified Waspaloy, the creep rates and, hence, the creep resistance varied with heat treatment and test conditions in similar manner to the rupture strengths.

Fracture Characteristics

The fracture characteristics of Modified Waspaloy were similar to those previously reported for Waspaloy [1]. Both smooth and notched specimens failed by initiation and relatively slow growth of intergranular cracks followed by transgranular fracture. The latter fracture occurred when the increase in stress on the load bearing area, due to growth of the intergranular crack, exceeded that necessary to cause rapid shear. The intergranular and transgranular parts of the fracture were readily distinguishable by visual examination. (The lengths of the intergranular cracks, expressed as a percentage of the specimen width, are included in Table 2.)

Optical metallography showed that the fractures of tensile and creep-rupture tested specimens of TD-Ni,Cr were also both transgranular and intergranular. Intergranular cracks were distributed across the fractures. In notched specimens, the fracture adjacent to one, or both notches, was intergranular. The total intergranular crack length increased with decreasing stress and increasing temperature. Extensive intergranular cracking, perpendicular to the loading direction, occurred adjacent to the fractures and throughout the gauge sections of the smooth specimens (Figure 5). Intergranular cracks were also found in several test specimens discontinued before rupture. These results indicate that failure occurred by intergranular crack initiation and growth, the linking of microcracks, and finally transgranular fracture.

Stress Relaxation and Creep Characteristics

Previous studies have shown that notch sensitive behavior is dependent on the manner which stress concentrations, introduced by the edge-notches, are relaxed [1,3]. When notched specimens are tested at stresses above the approximate 0.2 percent offset yield strength, yielding on loading reduces the stresses across the specimen at the base of the notch to the approximate nominal stress. Under these conditions, time-dependent notch sensitivity has not been observed. When notched specimens are loaded below the yield stress, the stress concentrations can only be fully relaxed by creep (from the approximate yield strength to the nominal stress). A material exhibits time-dependent notch sensitivity when the creep deformation necessary to relax stress concentrations causes excessive damage resulting in premature crack initiation. Experimentally, time-dependent notch sensitivity has been shown to occur when sharp edged-notched sheet specimens were loaded below the approximate 0.2 percent offset yield strength and when tests on smooth specimens showed that 0.1 and 0.2 percent creep consumed large fractions of rupture life [1,3]. Creep data for TD-Ni,Cr and Modified Waspaloy were evaluated to determine whether this same correlation applied:

(1) Analysis of the data for TD-Ni,Cr showed that within the range of test data at 1000° and 1200°F (538°,649°C) the life fractions for 0.1 and 0.2 percent creep were at low levels (Figure 6). Thus, the iso-creep strain characteristics were consistent with the fact that no time-dependent notch sensitivity occurred. At the higher temperatures and lower stresses, large life fractions were utilized for small creep strains. Despite this, no time-dependent notch sensitivity was observed. This apparent contradiction of the correlation was due to "negative creep" which is considered in the following section.

(2) For Modified Waspaloy, the life fractions for small amounts of creep were generally at very low levels (Figure 7). Correspondingly, no time-dependent notch sensitivity was observed (Figure 3). (At 1300°F, somewhat larger life fractions were consumed for 0.1 and 0.2 percent creep due to "negative creep.")

Negative Creep

When exposed under stress at elevated temperatures, many nickel-base superalloys shrink, i.e , exhibit "negative creep." Creep-rupture tests are usually conducted at test conditions for which the amount of positive creep deformation is larger than the negative creep. As a consequence, the measured deformation-time characteristics appear normal. In the present investigation, for both materials tested at the higher temperatures and lower stresses, the amount of positive deformation was relatively small compared with the negative creep. As a result, negative minimum creep rates were observed (Tables 1, 2, and Figure 8) and, large life fractions were utilized for small creep strains (Figure 6).

There is evidence that indicates that negative creep is probably due to an ordering of the Ni-Cr lattice [6]. Being a volumetric charge, it should not act to relax stress concentrations or influence the notch sensitive behavior. Results for TD-Ni,Cr tests, for which the creep rate was expected to be low, showed that shrinkages of at least 0.2 percent can occur. It is therefore probable that the apparent contradiction between creep characteristics and notch sensitive behavior was due to the influence of negative creep on the observed deformation-time curves.

MICROSTRUCTURAL FEATURES

TD-Ni,Cr and Modified Waspaloy were studied in the as-heat treated conditions in order to characterize the microstructural features present. Subsequently, tensile and creep-rupture tested specimens were examined primarily to establish the dislocation motion mechanisms. Samples were taken somewhat removed from the fractures of smooth specimens and from the shoulders of notched specimens. The latter specimens provided samples exposed at relatively low stresses (0.7 times the applied stress).

TD-Ni,Cr

As-Received Material ("Original Condition")

The average grain diameters were 0.16 mm and 0.08 mm in the longitudinal and transverse directions, respectively (Figure 9a). The ThO_2 particles ranged in size from about 100 to 1000 Å with a mean of approximately 285 Å (Figure 9b). A negligible number of randomly distributed dislocations were present. Many annealing twins (about 0.2μ wide) were observed (Figure 9b).

Tested Specimens

In specimens tensile tested at 1000°, 1400°, and 1800°F, (538°, 760°, and 982°C) many elongated dislocation loops were observed pinned to ThO_2 particles (Figure 10a). These are prismatic loops which do not lie in the original slip lane and indicate that the dislocations by-passed the oxide particles by cross slip [7]. The dislocation density decreased with increasing test temperature (decreasing elongation at fracture).

The microstructures (Figure 10b) of specimens creep-rupture tested at 1000°F (538°C) and at 1400°F (760°C) at 28.6 ksi (197 MN/m^2) also indicated that cross-slip had occurred.

In specimens creep-rupture tested at the higher temperatures and lower stresses, prismatic dislocation loops were not observed (Figure 10c). In these cases the dislocations by-passed the ThO_2 particles by climb.

For a number of tests at high stresses at 1200°, 1400°, and 1600°F (649°, 760° and 871°C) only a limited number of prismatic dislocation loops were observed. It was concluded that dislocations by-passed the oxide particles by both climb and cross slip. The minimum creep rate characteristics previously reported (Figure 4), indicated that climb was the predominant mechanism.

In all tensile and creep-rupture tests, the deformation was homogeneous. The dislocation mechanisms observed, as a function of test stress and temperature, are summarized in Figure 11.

Modified Waspaloy

As-Heat Treated

Limited examination of the as-heat treated materials indicated that the microstructural features, other than the γ' morphology, were similar to those previously reported from similarly heat treated Waspaloy [3]. The average grain size was 0.036 mm (Figure 12). The carbides present were identified, by selected area electron diffraction, to be Ti(C,N), and $M_{23}C_6$. The former carbide was a randomly distributed globular precipitate. The grain boundaries were partially filled with $M_{23}C_6$ carbide. A number of twin boundaries, particularly twin ends, also contained $M_{23}C_6$. As was the case for Waspaloy, γ' was present as coherently precipitated spherical particles. Due to lower aluminum and titanium additions, the γ' volume fraction was less than for Waspaloy (0.095 compared to 0.235).

The aging treatments, 3 hr at 1325°F (718°C), 1 hr at 1400°F (760°C), and 16 hr at 1400°F (760°C) resulted in γ' particles approximately 40, 75, and 140 Å in diameter, respectively. No zones depleted of γ' occurred adjacent to grain boundaries.

Tested Specimens

In tensile and creep-rupture tests at 1000° and 1100°F (538° and 593°C), limited γ' growth occurred during the test exposures. Dislocations either sheared or cross slipped around the relatively small particles. In smooth specimens tested at the higher stresses, prismatic dislocation loops were observed indicating that dislocations by-passed the γ' by cross-slip (Figure 13a). Under these conditions, the deformation was homogeneous. In samples (including these from the shoulders of notched specimens) exposed at lower stresses, superdislocations were observed (Figure 13b). A few dislocations were also present as extended stacking fault ribbons. These observations demonstrate that dislocations sheared the γ' particles. When this mechanism occurred, the deformation was localized in slip bands. At intermediate stresses, both dislocation mechanisms contributed to the deformation.

During the higher temperature creep-rupture exposures, considerable γ' growth occurred. In a number of test specimens for which the particles were large, dislocations were observed bowing between particles leaving pinched off concentric dislocation loops (Figure 13c). In other specimens, it was evident that the dislocations by-passed the γ' by both cross slip and by looping. In all cases the deformation was homogeneous.

The variations in dislocation mechanism observed with changes in test stress and γ' particle size are presented in Figures 14 and 15.

DISCUSSION

TD-Ni,Cr

Theories have been proposed to explain the manner in which edge and screw dislocations can by-pass particles by cross-slip [8]. Experimentally, cross-slip has been shown to occur for a number of dispersion hardened systems [9], the majority of which have high stacking fault energies. Cross-slip has been shown to occur for TD-Ni [10]. The stacking fault energy of nickel is lowered by the addition of chromium. (30 weight percent chromium decreases the stacking fault energy from about 225 to 70 erg/cm^2—ref. 11.) Consequently, cross slip could be expected to be relatively difficult in TD-Ni,Cr. Nevertheless, it does occur in low temperature, high stress, tensile and creep-rupture tests.

At high temperatures and low stresses, dislocations by-pass ThO_2 particles by climb. At each temperature there is a critical resolved shear stress, CRSS, above which the dislocations do not climb over the dispersed particles, but by-pass them by cross-slip (or looping). This concept was first considered theoretically by Orowan [12]. The following formulation for the "Orowan Stress" (the increase in CRSS due to the presence of non-deforming particles) was subsequently reported by Ashby [13]:

$$\Delta\tau_o = B \frac{A\mu b}{2\pi(\lambda - d)} \ln \frac{d}{4b} \quad , \qquad (1)$$

where μ is the matrix shear modulus, b the Burger vector, γ the mean planar center-to-center particle separation, d the mean planar particle size (0.82 x mean particle size, D) and B a statistical factor (about 0.85) related to the occurrence of a distribution of interparticle spacings. A is a constant equal to 1 for an edge dislocation and $(1-\nu)^{-1}$ for a screw dislocation, where ν is Poisson's ratio.

Several relationships have been proposed to permit calculation of λ values from the precipitate volume fraction V_f and the mean particle size [14]. These are based on assumed arrangements of the particles in space.

Simple cubic: $$\lambda = d\ (\pi/4V_f)^{1/2} \quad . \tag{2}$$

Face centered cubic: $$\lambda = d\ (1/V_f)^{1/2} \quad . \tag{3}$$

Random: $$\lambda = d\ (\pi/16V_f)^{1/2} \quad . \tag{4}$$

The results of the microstructural study of TD-Ni,Cr established the "Orowan Stress" as a band of values (Figure 11). The minimum creep rate characteristics (Figure 4) indicated that the upper curve probably best represents the stresses where the creep controlling mechanism changes from climb to cross-slip. These experimentally determined values were compared with those calculated using equation (1). The constant A was assumed to be 1.2 (the mean value for edge and screw dislocations). Values for the shear modulus, derived from tensile data were: 0.8, 0.7, and $0.5 \cdot 10^7$ psi (5.5, 4.8, and $3.4 \cdot 10^4$ MN/m^2) at 1000°, 1400° and 1800°F (538°, 760°, and 982°C), respectively. The Burger vector was 2.5 Å and the ThO_2 volume fraction 0.0174. Poisson's ratio was assumed to be 0.30. The CRSS values calculated were multiplied by the Taylor factor [15] so that they could be compared directly with the "applied stress" values determined experimentally. The factor of 3.06 which was used can be considered to be an upper bound for this "stress conversion." The results (Table 3) show that the theoretical values vary by a factor of about 2, depending on the equation, (2)-(4), used to calculate the center-to-center particle separation. The experimentally determined stress values are within the range of those determined theoretically. The ThO_2 dispersion should almost certainly be described as random.

Theoretical stresses based on this assumption were in error by a factor of about 2. Considering the approximations involved, this agreement is surprisingly good.

The ThO_2 particles contribute to the creep resistance of TD-Ni by impeding dislocation motion. It has been proposed, however, that the good creep characteristics of this alloy are primarily attributable to the fine grain size and cellular substructure formed during fabrication and stabilized by the oxide particles [10]. The creep resistance at 1000°F for the TD-Ni,Cr studied was similar to that reported for TD-Ni [10]. However, the creep resistance of TD-Ni,Cr decreased much more rapidly with increasing temperature than was the case for TD-Ni. Correspondingly, the microstructures of the as-received TD-Ni,Cr did not reveal any substructure (consistent with the relatively low stacking fault energy of this material). In addition, the grain size of TD-Ni,Cr was about 10 times that of TD-Ni (80-160μ compared to 1-2μ). Thus in contrast to TD-Ni, the ThO_2 particles in TD-Ni,Cr increased the creep strength only by providing resistance to dislocation motion.

Minimum creep rate characteristics are often represented by the familiar expression:

$$\dot{\varepsilon}_m = A(t)\sigma^n \quad , \tag{5}$$

where n determines the overall stress dependence of the creep process. For TD-Ni,Cr, n was 10 at 1000°F (538°C) and about 30 at higher test temperatures. These values are significantly greater than predicted by theories for cross slip and climb (about 5). Anomalously high values for n, reported by other authors for dispersion hardened materials, have been explained in terms of "internal stress" and/or grain orientation effects [10,11,16,17]. "Negative creep" that occurs for TD-Ni,Cr reduced the minimum creep rates, particularly at the lower test stresses, and thereby must have acted to increase the measured n values.

Modified Waspaloy

For Modified Waspaloy ($V_f = 0.095$), γ' particles were by-passed by cross-slip and by looping. For TD-Ni,Cr ($V_f = 0.017$) only cross-slip occurred, whereas, for Waspaloy ($V_f = 0.23$) only the looping mechanism was observed [3]. These results suggest that increasing the volume fractio of precipitate tends to change the by-pass mechanism from cross-slip to looping. Other variables such as the stacking fault energy and test temperature, can also be expected to influence this transition.

Both cross-slip and looping are dependent on the ability of dislocations to bow between precipitate particles. Consequently, they occur at stresses greater than the "Orowan stress." For Modified Waspaloy, both the average γ' size and the test temperature varied. To simplify evaluation of the experimental results, the data were compensated for temperature variation using the following relationship derived from equation (1):

$$\sigma_{1000^\circ F} = \sigma_T \frac{\mu_{1000^\circ F}}{\mu_T} \quad . \tag{6}$$

The dislocation mechanisms observed are presented in Figure 14 as a function of stress compensated to 1000°F (538°C) and the mean γ' particle size.

Orowan stresses at 1000°F were calculated using equation (1) in a similar manner described for TD-Ni,Cr. In this case, the following values were used: $\mu = 1.02 \times 10^7$ psi (7.03×10^4 MN/m^2), b = 2.52 Å and, $V_f = 0.095$. Periodic or "ordered" precipitate distribution are observed in many superalloys. These structures occur when the γ' particles are large and/or the γ'/γ lattice mismatch is large [18]. Based on the composition of Modified Waspaloy and the observed spherical γ' morphology, it can be concluded that the γ'/γ mismatch was relatively small (similar to Waspaloy which has a mismatch of about 0.3%). Also, the particles in the as-heat treated materials were relatively small. Consequently, the γ' precipitate particles were probably "randomly" distributed. Theoretical values for the Orowan stress, using this assumption, differ by a factor of about 2 from the experimental values (Figure 14). It should be considered coincidental that the experimental values agree well with the theoretical stresses based on face centered cubic and simple cubic distributions.

Because the γ' is a coherent precipitate, it can be sheared by dislocations. Gleiter and Hornbogen [18] reported that the increase in CRSS due to ordered precipitate particles sheared by dislocations, $\Delta\tau_{sh}$, is given by the following equation:

$$\Delta\tau_{sh} = 0.28\, \gamma_A^{3/2}\, V_f^{1/3}\, (D/2)^{1/2}\, \mu^{-1/2}\, b^{-2} \qquad , \tag{7}$$

where γ_A is the antiphase boundary energy. $\Delta\tau_{sh}$ values at 1000°F (538°C), were calculated using a value of 260 erg/cm^2 for γ_A [19]. To permit comparison with the microstructural observations (Figure 15), the test stresses were compensated to 1000°F (538°C) using the following relationship from equation (7):

$$\sigma_{1000°F} = \sigma_T \left(\frac{\mu_T}{\mu_{1000°F}}\right)^{1/2} \tag{8}$$

All of the tests stresses (Figure 15) were greater then the calculated values ($\Delta\tau_{sh}$ x Taylor factor, 3.06). The specimens tested at stresses slightly greater than the calculated values had very low dislocation densities. (Below $\Delta\tau_{sh}$, the only mechanism that can occur is climb, which due to the relatively low test temperatures, should contribute very little to the deformation.) Therefore, it can be concluded that the theoretical values for $\Delta\tau_{sh}$ are in very good agreement with the experimental results.

The CRSS for shearing increases with $D^{1/2}$ (equation (7)) while for cross slip or looping it decreases as D increases (equation (1)). Therefore, for each γ' strengthened alloy (constant V_f), there is a critical particle size at which the dislocation mechanism changes from shearing to by-passing the precipitate particles. This is shown for Modified Waspaloy in Figure 16.

Correlation of the Time-Dependent Edge-Notch Sensitivity with the Dislocation Mechanism

In previous studies, a correlation was established between the dislocation motion mechanism and the time-dependent notch sensitive behavior [2,3]. Precipitate particles, γ' in Waspaloy and γ' and γ''-Ni_3Cb (bct) in Inconel 718, smaller than the critical size were sheared by dislocations. Under these conditions, the deformation was localized and time-dependent notch sensitivity occurred. Particles larger than the critical size were by-passed by dislocations, the deformation was homogeneous and no time-dependent notch sensitivity was observed.

The results of the presently reported investigation are consistent with the above correlation. For TD-Ni,Cr, the dislocations by-passed the ThO_2 particles for all test conditions. Correspondingly, time-dependent notch sensitivity did not occur. In a number of notched specimen tests for Modified Waspaloy at the lower test temperatures, dislocations sheared the γ' particles (Figure 13b). However, no time-dependent notch sensitivity was observed. This occurred because the test stresses were above the yield stresses. For notched specimens tested below the yield strength, either the test times were extremely long or the test temperatures were relatively high. As a result, considerable γ' growth occurred during the test exposures. This caused the dislocation mechanism to change from shearing to by-passing which reduced the susceptibility to time-dependent notch sensitivity so that none was observed. Thus time-dependent notch sensitivity did not occur for Modified Waspaloy due to the yield strength and γ' growth rate characteristics.

The yield strength, σ_y, of a dispersion hardened alloy can be expressed by the following relationship:

$$\sigma_y = \sigma_m + 3.06 \, \Delta\tau_p \quad , \tag{9}$$

where σ_m = yield stress of the matrix, $\Delta\tau_p$ is the increase in CRSS due to the presence of the particles ($\Delta\tau_o$ or $\Delta\tau_{sh}$). Consideration of the yield strength in this manner (Figure 16) permits a regime of test stress and particle size to be defined for which time-dependent notch sensitivity can be expected.

Rapid growth of γ' in Modified Waspaloy prevented time-dependent notch sensitivity from occurring at the low test stresses. The γ' ripening rate decreases considerably with increasing volume fraction [20]. For alloys with higher γ' volume fraction than Modified Waspaloy, particle growth cannot be relied upon to prevent notch sensitive behavior from occurring. Heat treatments should be selected to produce γ' particles larger than the critical size. Under these circumstances, it is actually desirable to limit the growth rate (by compositional control) so as to reduce strength degeneration that will accompany γ' growth.

Hardness tests or yield strengths reflect the variations in CRSS with γ' size and can, therefore, be used in the selection of heat treatments. For Waspaloy [3] and Inconel 718 [2] room temperature hardnesses were correlated with the notch sensitive behavior. For Modified Waspaloy (Figure 17), hardness tests correctly indicated that γ' particles in the as-heat treated materials to be smaller than the critical size (maximum hardness). It should be recognized that the critical size varies somewhat with temperature. This arises because of changes in the shear modulus, μ, with temperature. From the equations presented ((1), (4), and (7)) it is evident that as μ decreases with temperature the critical size decreases with increasing temperature. Thus if a heat treatment is selected, by room temperature hardness tests, so that the γ' particles are larger than the critical size they will also be larger than the critical size at elevated temperatures.

SUMMARY OF RESULTS

A research program was carried out to determine whether an oxide dispersion hardened alloy, TD-Ni,Cr, and a low volume fraction γ' strengthened nickel-base alloy, Modified Waspaloy, were susceptible to time-dependent edge-notch sensitivity. The results were evaluated in terms of the mechanical characteristics of the alloys and the dislocation motion mechanisms operative.

Time-dependent notch sensitivity was not observed for 0.015-in. (0.38 mm) thick, stress relieved, TD-Ni,Cr sheet at temperatures from 1000° to 1800°F (538°-982°C). Dislocations by-passed ThO_2 particles by cross-slip at high test stresses and low temperatures and by climb at low test stresses and high temperatures. The stresses at which the mechanism changed from climb to cross-slip (Orowan stresses) were in reasonable agreement with theoretical predictions.

Time-dependent notch sensitivity was not evident from tests of 0.025-in. (0.64 mm) thick Modified Waspaloy sheet at temperatures from 1000° to 1400°F (538°-760°C). In tests at relatively low stresses, dislocations sheared γ' particles smaller than a critical size (about 180Å); larger particles were by-passed by cross-slip or looping. At stresses higher than the Orowan stress, small γ' particles were by-passed by cross-slip. Results indicated that the dislocation mechanisms observed can be usefully evaluated in terms of theoretical concepts.

As far as could be determined, the results of the investigation were consistent with the following important concepts developed for Waspaloy [1,3] and Inconel 718 [2]: (a) Time-dependent edge-notch sensitivity occurs when notched specimens are loaded below the approximate 0.2 percent smooth specimen offset yield strength and when data from smooth specimens indicate that small amounts of creep consume large rupture life fractions. (b) When precipitate particles are sheared by dislocations, the deformation is localized and time-dependent notch sensitivity occurs. When dislocations by-pass precipitate particles the deformation is homogeneous. Under these conditions, no time-dependent notch sensitivity has been observed.

To date, alloys have been studied which have coherent and noncoherent precipitate volume fractions ranging from 0.017 to 0.23. The applicability of the above correlations for these alloys suggests that they can be used to characterize the notch sensitive behavior of an even wider range of superalloy sheet materials.

It is apparent that hardness tests can be used to select heat treatments that produce γ' particles larger than the critical size and, therefore, avoid time-dependent notch sensitivity.

REFERENCES

1. Wilson, D. J. "Sensitivity of the Creep-Rupture properties of Waspaloy Sheet to Sharp-Edged Notches in the Temperature Range 1000°-1400°F, Trans. ASME, J. Basic Eng. 94 (1972), No. 1, pp. 13-21.

2. Wilson, D. J. "Relationship of Mechanical Characteristics and Microstructural Features to the Time-Dependent Edge Notch Sensitivity of Inconel 718 Sheet" (accepted for publication to ASME).

3. Wilson, D. J. "The Dependence of the Notch Sensitivity of Waspaloy at 1000°-1400°F on the Gamma Prime Phase," Trans. ASME, J. Basic Eng. (In print).

4. Bigelow, W. C., J. A. Amy, and L. O. Brockway. "Electron Microscope Identification of the Gamma Prime Phase of Nickel-Base Alloys," Proc. ASTM 56 (1956), pp. 945-953.

5. Kriege, O. H., and J. M. Barris. "The Chemical Partitioning of Elements in Gamma Prime Separated from Precipitation Hardened Nickel-Base Alloys," Trans. ASM 62 (1969), pp. 195-200.

6. Nordheim, R., and N J. Grant. "Resistivity Anomalies in the Nickel-Chromium System as Evidence of Ordering Reactions," JISI 21 (1954), pp. 440-444.

7. Humphreys, F. J., and J. W. Martin. "The Deformation of Single Crystals of Copper and Copper-Zinc Alloys Containing Alumina Particles (II) Microstructural and Dislocation-Particle Interactions," Proc. Roy. Soc. (London) A318 (1970), pp. 73-92.

8. Hirsch, P. B., and F. J. Humphreys. "Physics of Strength and Plasticity." Mass. Inst. Tech. Press. (1969), pp. 189-216.

9. Duesbery, M. S., and P. B. Hirsch. "Fundamental Aspects of Dislocation Theory." N. B. S. Special Publication 317 (1969), pp. 1115-1134.

10. Wilcox, B. A., and A. H. Claurer. "Creep of Thoriated Nickel Above and Below 0.5 Tm," Trans. AIME 236 (1966), pp. 570-580.

11. Wilcox, B. A., A. M. Claurer, and W. S. McCain. "Creep and Creep Fracture of a Ni-20Cr-2ThO_2 Alloy," Trans. AIME 239 (1967), pp. 1791-1795.

12. Orowan, E. "Symposium on Internal Stresses in Metals and Alloys," JISI (1948), pp. 451-465.

REFERENCES (Concluded)

13. Ashby, M. F. "Physics of Strength and Plasticity," Mass. Inst. Tech. Press (1969), pp. 113-131.

14. Ashby, M. F. "The Hardening of Metals by Non-Deforming Particles," Z. Metallkunde 55 (1964), pp. 5-17.

15. Kocks, U. F. "The Relation Between Polycrystal Deformation and Single Crystal Deformation," Met. Trans. 1, No. 5 (1970), pp. 1121-1159.

16. Li, J.C.M. "Physics of Strength and Plasticity," Mass. Inst. Tech. Press (1969), pp. 245-253.

17. Claurer, A. H., and B. A. Wilcox. "Steady-State Creep of Dispersion Strengthened Nickel," Met. Sci. J. 1 (1967), pp. 86-90.

18. Gleiter, H., and E. Hornbogen. "Precipitation Hardening by Coherent Particles," Matls. Sci. Eng. 2 (1967-68), pp. 285-302.

19. Merrick, M. F. "The Structure of Nickel-Base Creep Resistant Alloys," Doctoral Thesis, University of Cambridge (1963).

20. Decker, R. F. "Strengthening Mechanisms in Nickel-Base Superalloys," presented at Steel Strengthening Mechanisms Symposium, Zurich, Switzerland (1964).

TABLE 1

SMOOTH AND NOTCHED ($K_t > 20$) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1800°F (538°- 982°C) FOR 0.015-INCH (0.38 mm) THICK TD-Ni,Cr SHEET

Smooth Specimens

Test Temperature (°F)	Test Temperature (°C)	Stress (ksi)	Stress (MN/m²)	Rupture Time (hr)	Elong. (%)	R.A. (%)	Minimum Creep Rate (%/hr)
1000	538	84.6	583	tensile[a]	9.8	16	--
		50	345	49.8	4.5	8	0.035
		40	276	628.8	4.4	6	0.0036
		36	248	2570.0	4.4	6	0.0010
		34	234	2473.0	4.2	8	0.0010
		30	207	9311.7	3.5	6	0.00029
1200	649	26	179	54.2	2.7	4	0.040
		24	165	351.1	2.4	6	0.0054
		22	152	9009	In Progress		0.00008
1400	760	39.5	272	tensile[a]	3.4	6	--
		28.6	197	0.5	2.7	6	--
		22	152	74.5	2.8	5	< 0.032
		20	138	973.7	2.3	4	0.00014
		16	110	11000	Discontinued		- ve[b]
1600	871	18	124	35.9	1.7	4	0.022
		16	110	132.1	2.5	2	0.0016
		14	97	3611.6	1.1	2	0.000050
		13	90	695.3	1.1	2	0.000004
		12	83	1944.0	1.6	2	- ve[b]
1800	982	21.6	149	tensile[a]	1.4	4	--
		12	83	47.7	---	2	0.18
		10	69	196.2	---	6	- ve[b]
		8	55	5183.6	---	-	- ve[b]

Notched Specimens

Test Temperature (°F)	Test Temperature (°C)	Stress (ksi)	Stress (MN/m²)	Rupture Time (hr)	N/S Strength Ratio
1000	538	65.6	452	tensile	.78
		40	276	31.1	.78
		34	234	803.6	.87
		30	207	2022.0	.88
1200	649	26	179	59.3	1.00
		24	165	708.0	1.02
1400	760	38.0	262	tensile	0.96
		20	138	839.8	1.0
		16	110	9062	Discontinued
1600	871	16	110	151.6	1.0
		14	97	501.7	1.0
		12	83	11000	In Progress
1800	982	23.3	161	tensile	1.08
		12	83	62.0	1.08
		10	69	2147.8	1.16

[a] 0.2% offset yield strengths:
1000°F (538°C) - 55.5 ksi (383 MN/m²)
1400°F (760°C) - 34.1 ksi (235 MN/m²)
1800°F (982°C) - 21.2 ksi (146 MN/m²)

[b] -ve = negative minimum creep

TABLE 2

SMOOTH AND NOTCHED ($K_t > 20$) SPECIMEN TENSILE AND CREEP RUPTURE PROPERTIES AT 1000° TO 1400°F (538°-760°C) FOR 0.025-INCH (0.64 mm) THICK MODIFIED WASPALOY SHEET SOLUTION TREATED AT 1/2 HOUR AT 1975°F (1080°C) AND AGED

Smooth Specimens									Notched Specimens						
Test Temperature		Stress		Rupture Time	Elong.	R.A.	Minimum Creep Rate	Intergranular Crack Length	Test Temperature		Stress		Rupture Time	Intergranular Crack Length	N/S Strength Ratio
(°F)	(°C)	(ksi)	(MN/m²)	(hr)	(%)	(%)	(%/hr)	(%)	(°F)	(°C)	(ksi)	(MN/m²)	(hr)	(%)	
Aged 3 Hours at 1325 F (718 C)															
1000	538	115.8	798	tensile*	54.0	40	--	0	1000	538	80.9	558	tensile	0	0.70
1100	593	80	552	171.9	15.8	25	0.020	19	1100	593	60	414	6743	In Progress	> 0.85
		70	483	2309.6	10.2	14	0.00034	30	1200	649	55	379	198.2	25	0.82
1200	649	70	483	135.4	9.2	16	0.015	23			45	310	5806	In Progress	> 0.87
		60	414	894.0	5.9	10	0.0012	28			40	276	2637	Discontinued	> 0.74
									1400	760	30	207	1155.8	63	~1.0
Aged 1 Hour at 1400 F (760 C)															
1000	538	116.5	804	tensile*	48.0	41	--	0	1000	538	80	552	1.0	0.5	~0.70
		95	655	4355.6	18.3	16	0.000090	--	1100	593	80	552	0.6	1	~0.70
1100	593	90	620	277.2	14.7	17	0.0018	10			60	414	9745	Discontinued	> 0.88
		80	552	1124.5	9.7	15	0.00044	19	1200	649	60	414	1357.0	48	~1.0
		70	483	5843.0	6.2	10	0.000080	40	1300	704	50	345	386.4	50	~1.0
1200	649	80	552	59.0	8.4	15	0.024	16							
Aged 16 Hours at 1400 F (760 C)															
1000	538	122.0	841	tensile*	50	41	--	0	1000	538	91.7	632	tensile	0	0.75
		110	758	310.5	25.8	25	0.015	4			85	586	5893.9	19	0.85
		100	689	4635.4	12.5	13	0.00040	13			70	483	4271	Discontinued	> 0.70
1100	593	100	689	205.0	16.5	19	0.029	7	1100	593	80	552	2679.5	24	0.98
		80	552	3542.5	4.7	12	0.00025	32			70	483	11184	Discontinued	> 0.96
1200	649	80	552	259.2	3.55	14	0.013	18	1200	649	60	414	770.3 ph	--	> 0.86
		60	414	2688.0			0.00029				55	379	7528.2	51	1.06
											45	310	8354	Discontinued	> 0.88
1300	704	50	345	351.4	6.2	13	- ve	42	1400	760	30	207	1170.8	47	1.15
1400	760	25	172	1592.1	15.5	28	0.00045	--							

Notes: ph = failed at pin hole. -ve - negative minimum creep rate.
*0.2% offset yield strengths at 1000°F:
3 hr at 1325°F (718°C) - 54.0 ksi (372 MN/m²)
1 hr at 1400°F (760°C) - 57.5 ksi (396 MN/m²)
16 hr at 1400°F (760°C) - 68.2 ksi (470 MN/m²)

TABLE 3

COMPARISON OF EXPERIMENTAL AND THEORETICAL VALUES OF THE OROWAN STRESS FOR TD-Ni,Cr

Temperature		Experimental Applied Stress		Theoretical Values* ($\Delta\tau_o$ x 3.06)					
				fcc		Simple Cubic		Random	
(°F)	(°C)	(ksi)	(MN/m^2)	(ksi)	(MN/m^2)	(ksi)	(MN/m^2)	(ksi)	(MN/m^2)
1000	538	30	207	20	141	23	162	57	392
1400	760	23	159	18	123	21	142	50	343
1800	982	16	110	13	88	15	101	36	245

*Theoretical values were calculated using three particle distributions: face centered cubic (λ = 1764 Å), simple cubic (λ = 1563 Å), and random (λ = 782 Å).

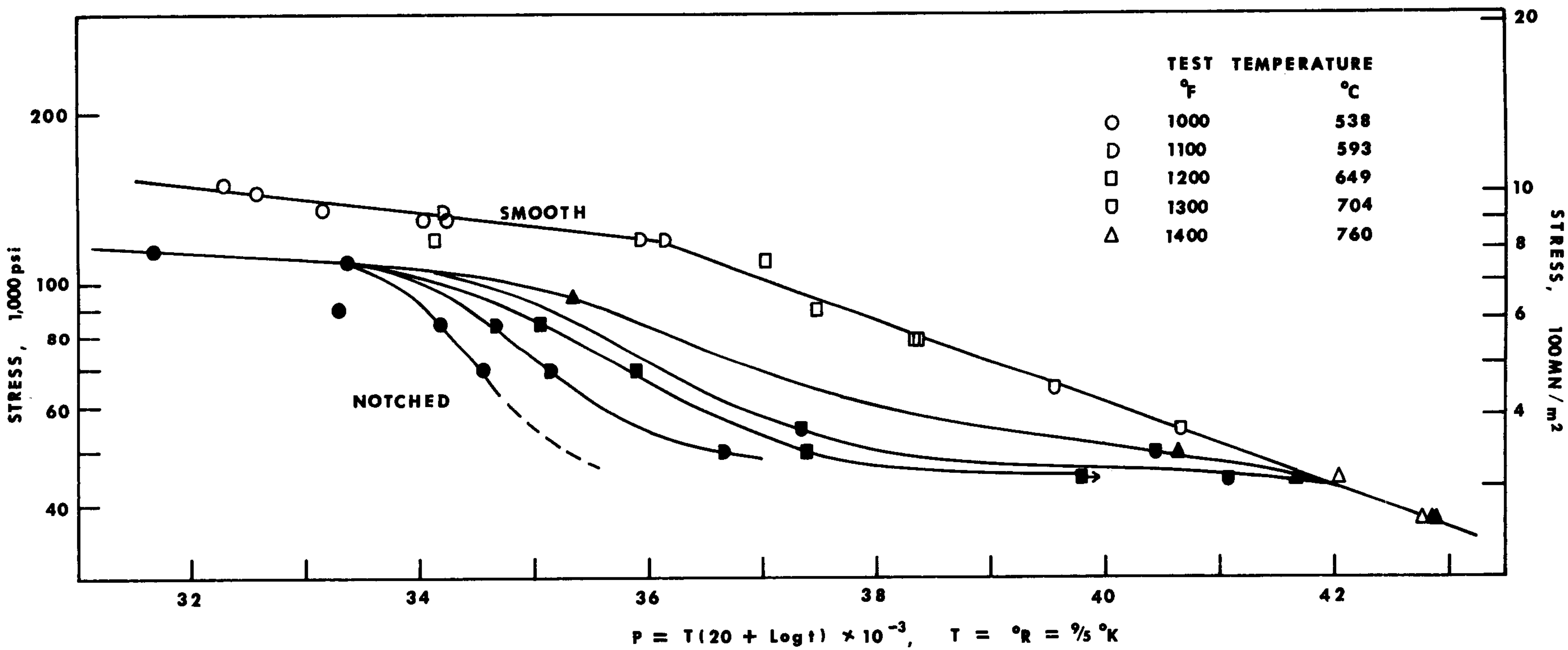


Figure 1. Larson-Miller parameter curves showing the time-temperature dependence of the rupture strengths of Waspaloy heat treated 1/2 hour at 1975°F (1080°C) and aged 16 hours at 1400°F (760°C). Reference 1

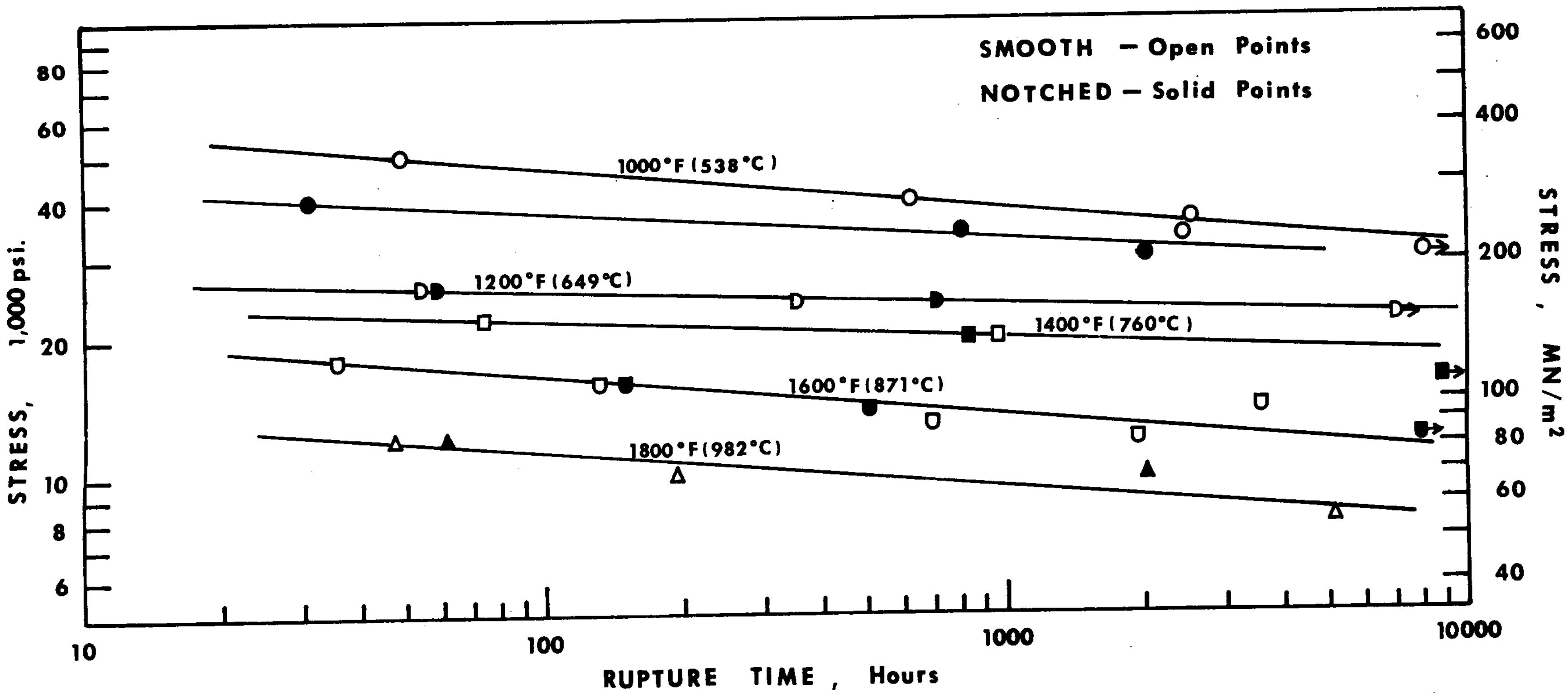


Figure 2. Stress versus rupture time data at temperatures from 1000° to 1800°F (538-982°C) obtained from smooth and notched specimens of 0.015-inch (0.38mm) thick, stress relieved, TD-Ni, Cr sheet. No time-dependent notch sensitivity was observed.

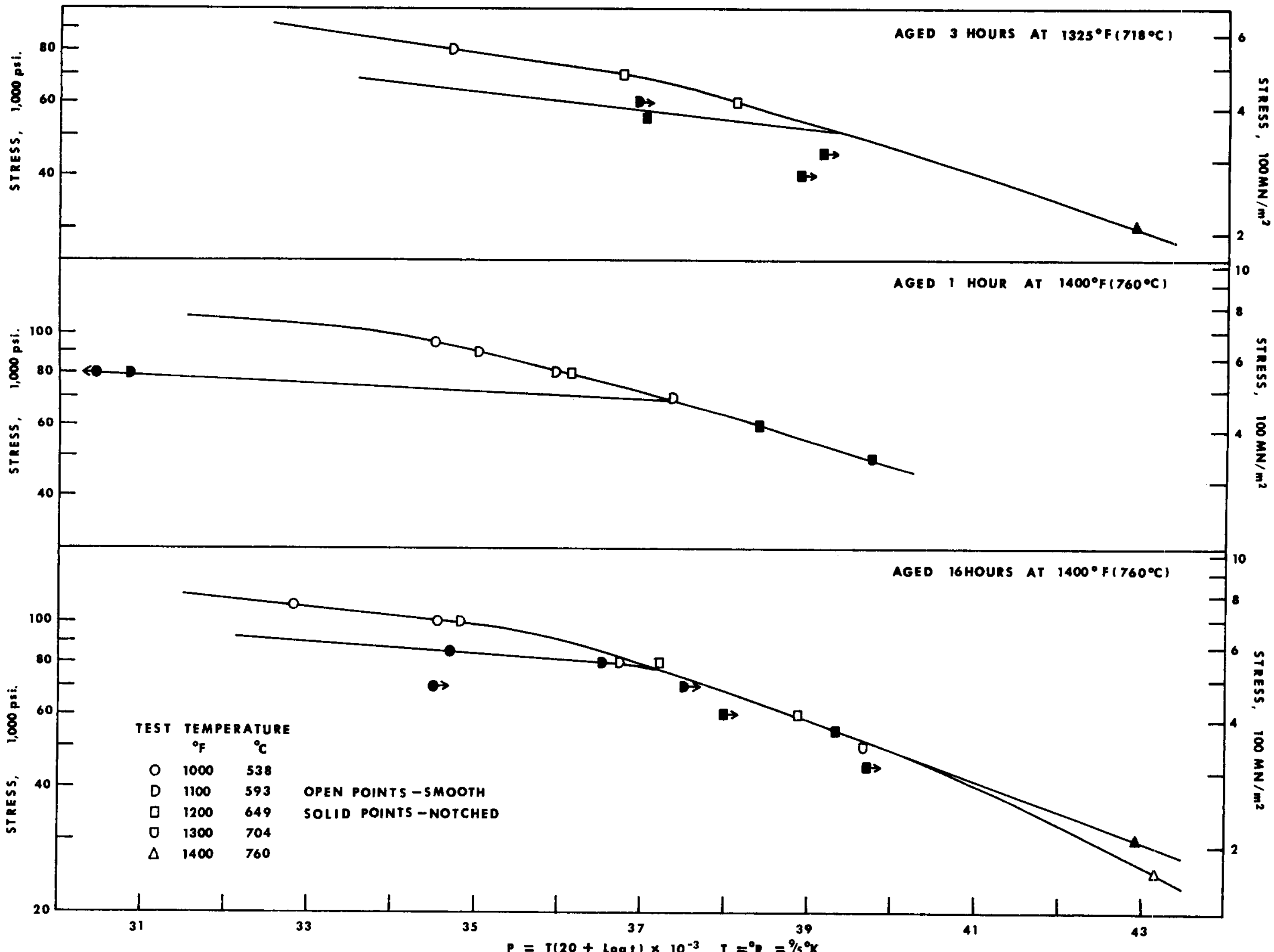


Figure 3. Larson-Miller parameter curves showing the time-temperature dependence of the rupture strengths of 0.025-inch (0.64mm) thick Modified Waspaloy sheet solution treated 1/2 hour at 1975°F (1080°C) and aged. Time-dependent notch sensitivity was not observed at test temperatures from 1000° to 1400°F (538-760°C).

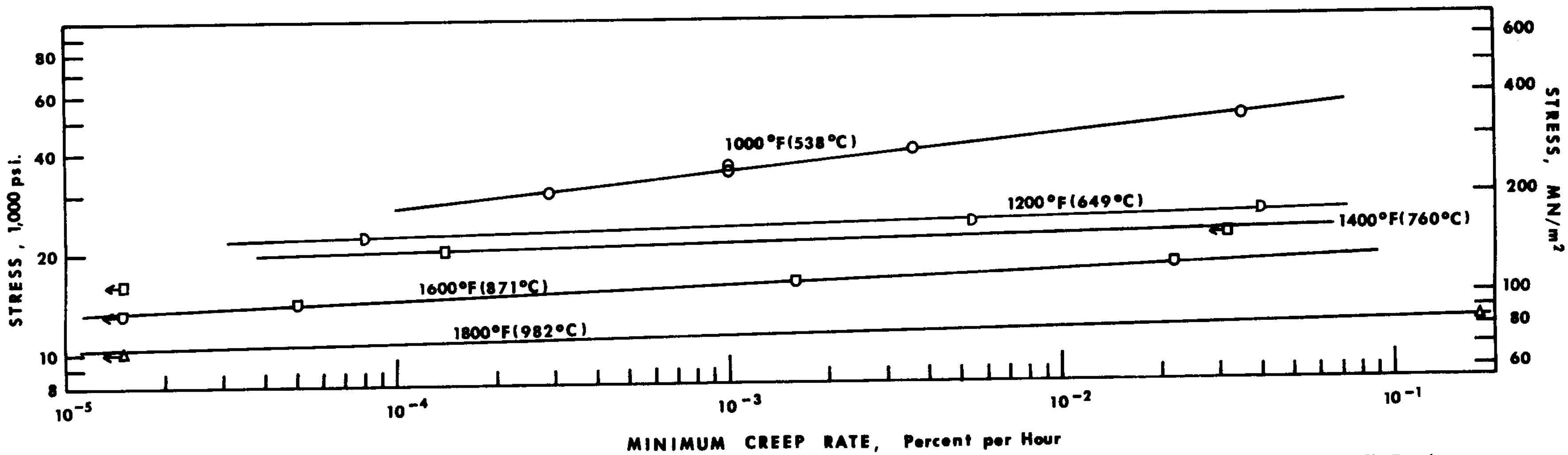
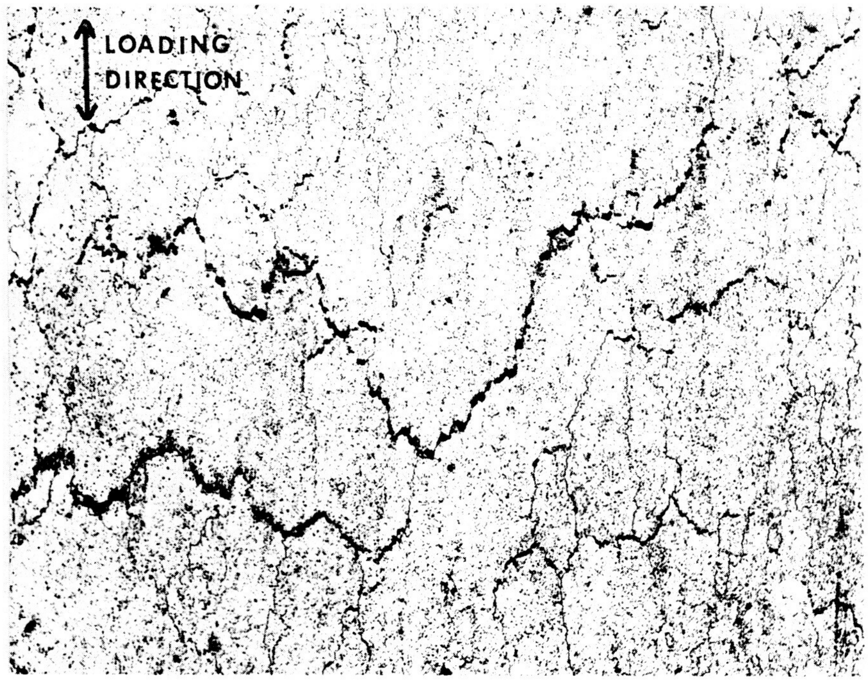


Figure 4. Stress versus minimum creep rate behavior at 1000° to 1800°F (538-982°C) for 0.015-inch (0.38mm) thick, stress relieved, TD-Ni, Cr sheet. The curve gradients indicate that the mechanism controlling creep changes between 1000°and 1200°F (538-649°C).



100x

Figure 5. Optical Photomicrograph showing subsidiary intergranular cracks in the gauge section of a smooth specimen of 0.015-inch (0.38mm) thick TD-Ni,Cr sheet creep-rupture tested at 12ksi. (82.7MN/m^2) at 1800°F (982°C).

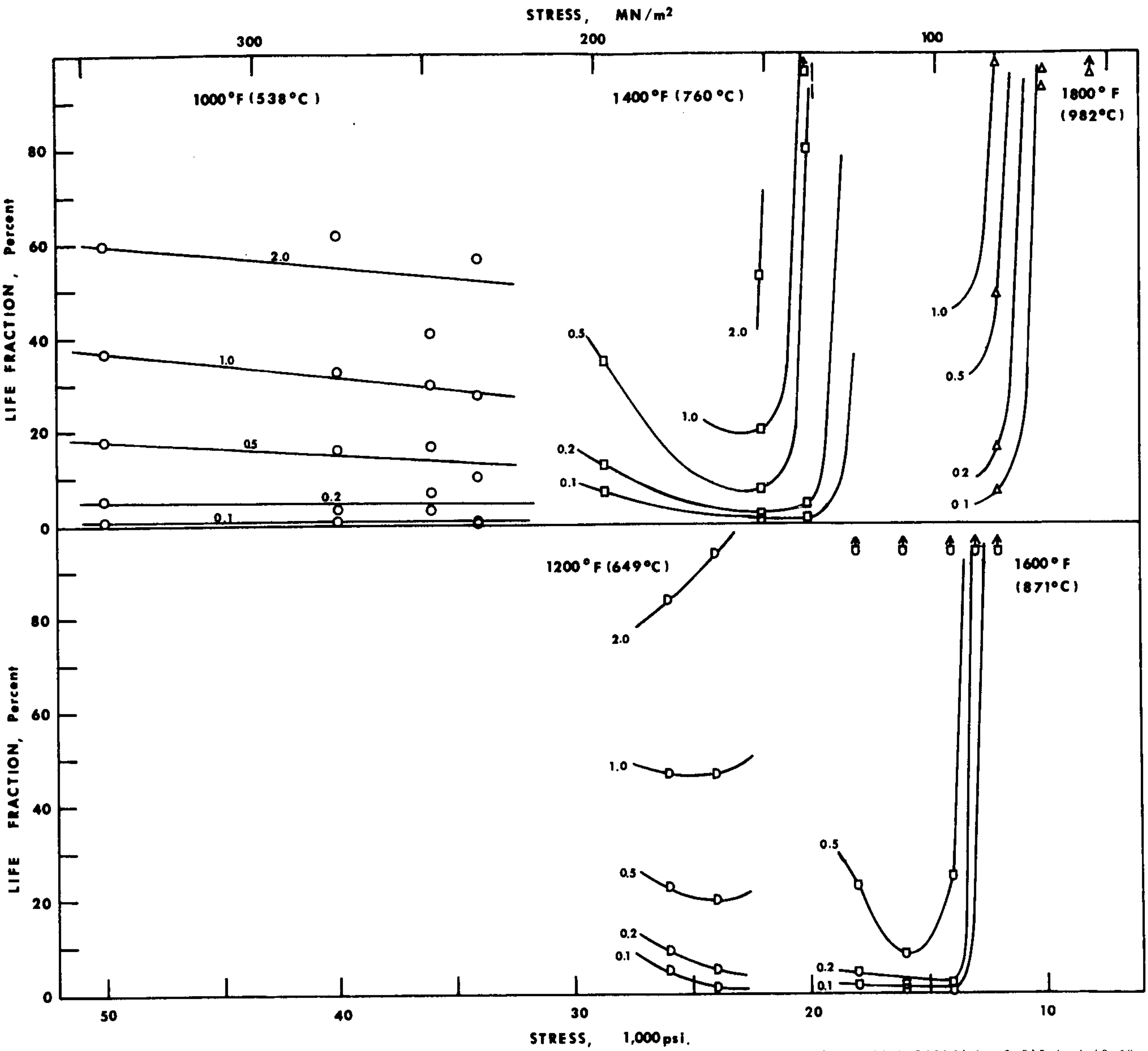


Figure 6. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1800°F (538-982°C) for 0.015-inch (0.38mm) thick, stress relieved, TD-Ni, Cr sheet. The apparant large amounts of rupture life consumed for small creep strain at the higher temperatures and lower stresses was due to "Negative Creep".

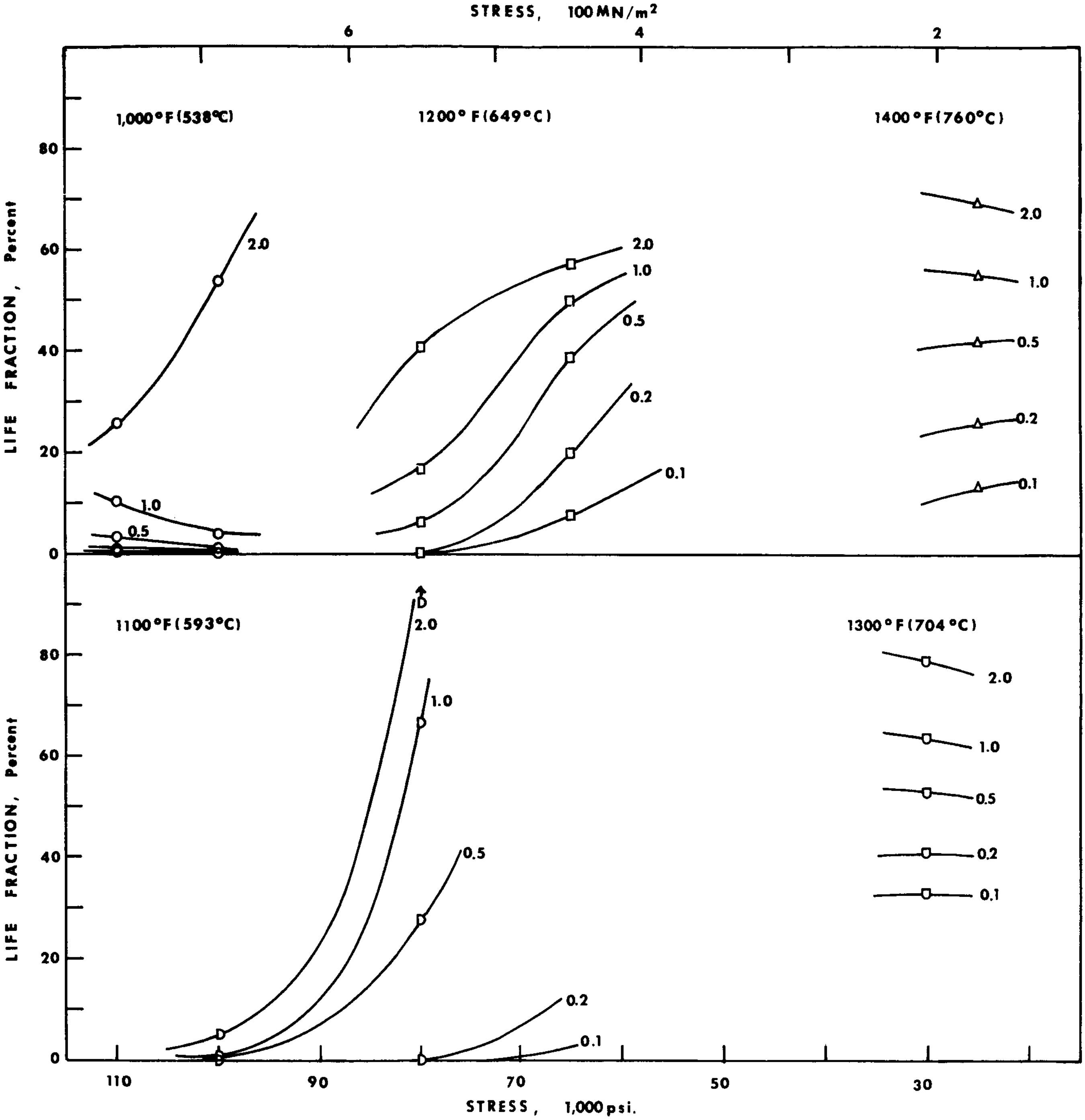


Figure 7. Iso-creep strain curves of life fraction versus stress at temperatures from 1000° to 1400°F (538-760°C) for 0.025-inch (0.64mm) thick Modified Waspaloy sheet heat treated 1/2hour at 1975°F (1080°C) plus 16 hours at 1400°F (760°C). Relatively little rupture life was consumed for small creep strains for all test conditions. Correspondingly, no time-dependent notch sensitivity was observed.

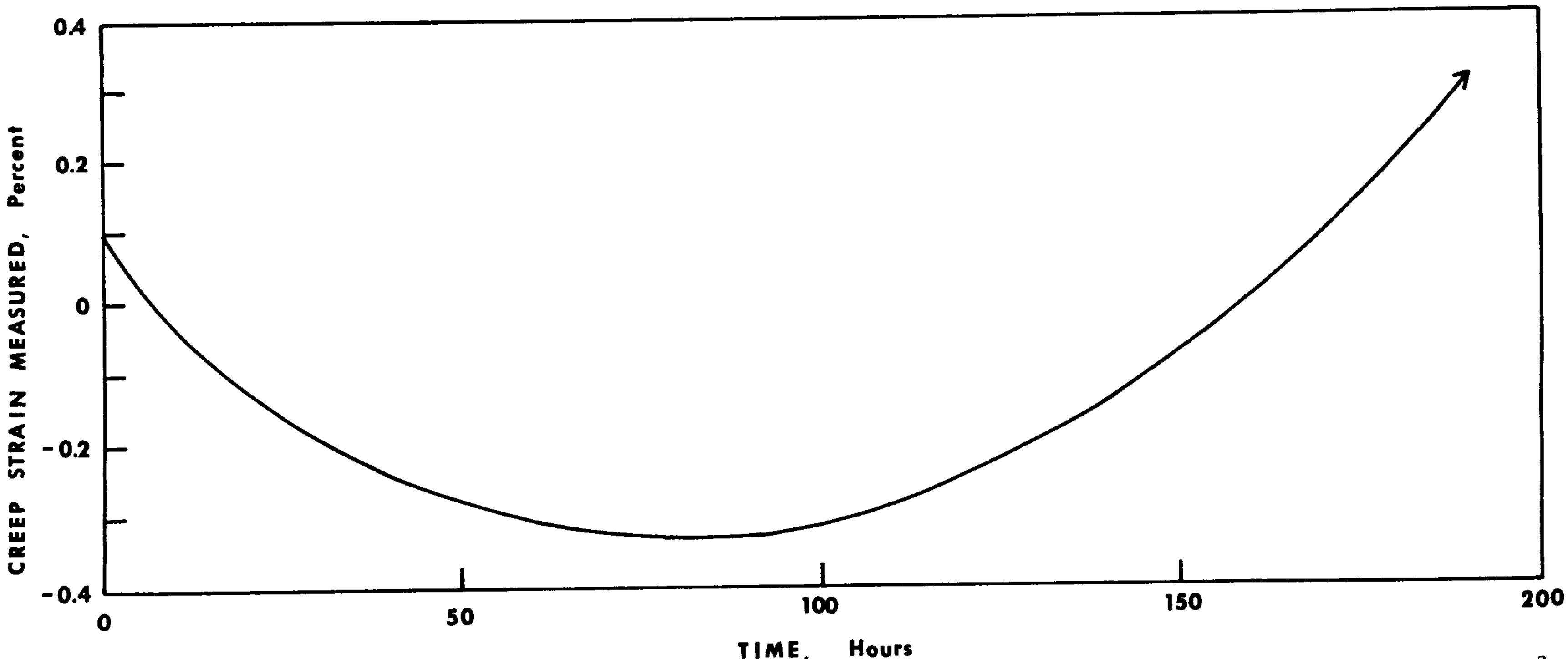
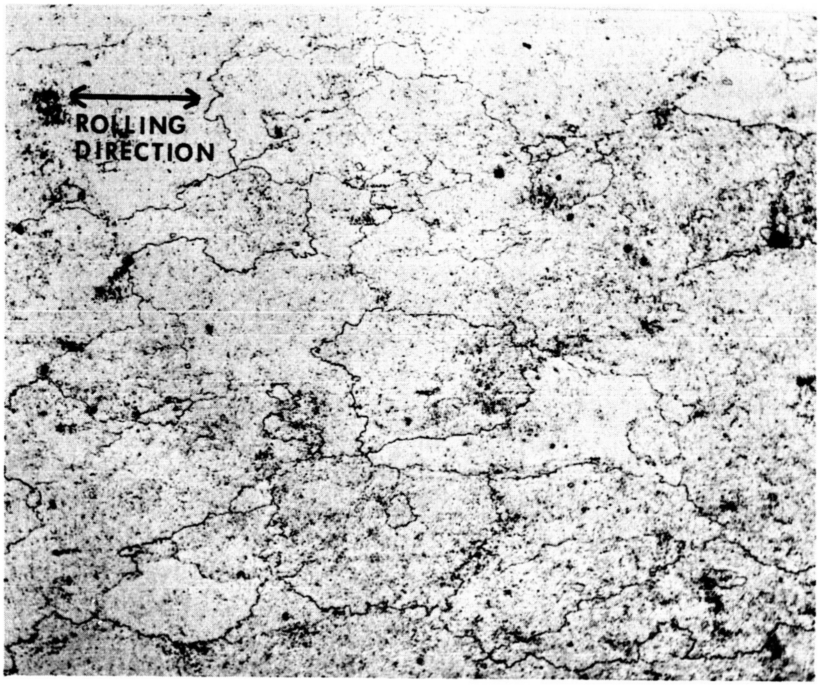
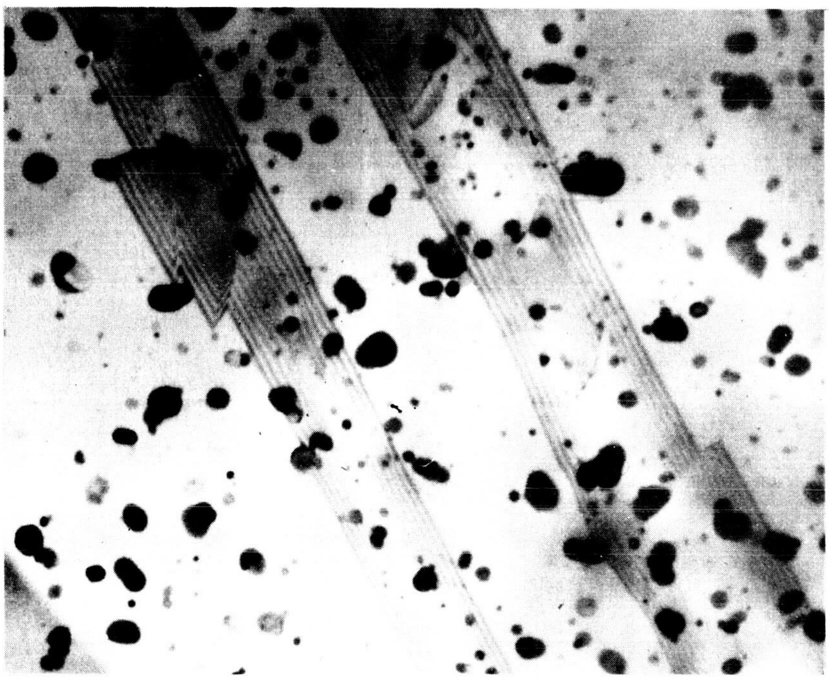


Figure 8. Deformation-time curve for a smooth specimen of TD-Ni, Cr sheet creep-rupture tested at 10ksi. (69 MN/m^2) at 1800°F (982°C). At these test conditions a large amount of "Negative Creep" occured resulting in a negative minimum creep rate.

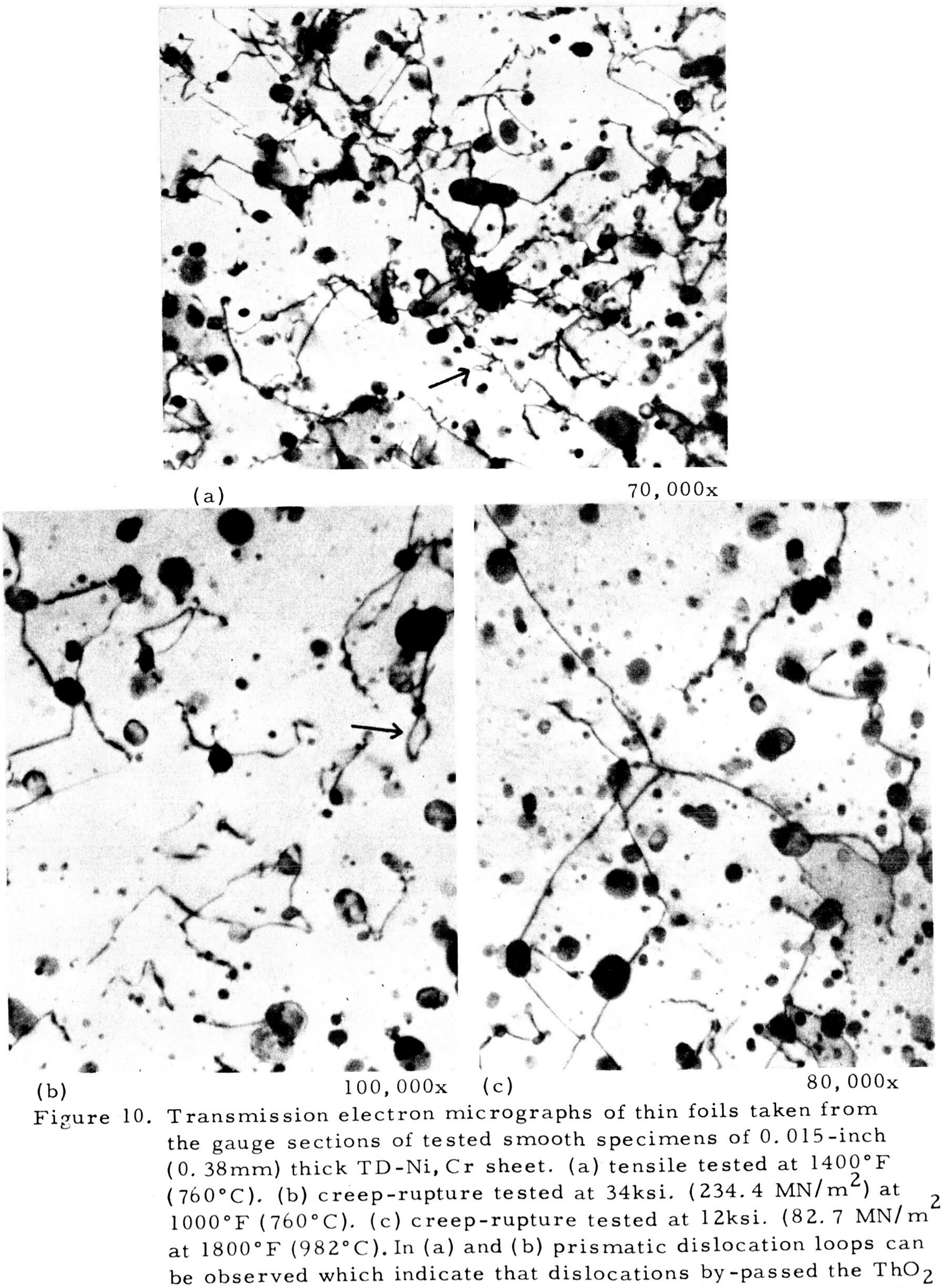


(a) 100x



(b) 50,000x

Figure 9. Optical and transmission photomicrographs of as-received 0.015-inch (0.38mm) thick, stress relieved, TD-Ni, Cr sheet The grains, 0.080- 0.16mm in diameter, were somewhat elongated in the rolling direction. Many narrow twins were present. Relatively few, randomly distributed, dislocations were observed.



(a) 70,000x

(b) 100,000x (c) 80,000x

Figure 10. Transmission electron micrographs of thin foils taken from the gauge sections of tested smooth specimens of 0.015-inch (0.38mm) thick TD-Ni, Cr sheet. (a) tensile tested at 1400°F (760°C). (b) creep-rupture tested at 34ksi. (234.4 MN/m^2) at 1000°F (760°C). (c) creep-rupture tested at 12ksi. (82.7 MN/m^2) at 1800°F (982°C). In (a) and (b) prismatic dislocation loops can be observed which indicate that dislocations by-passed the ThO_2 particles by cross-slip. Oxide particles in (c) were by-passed by climb.

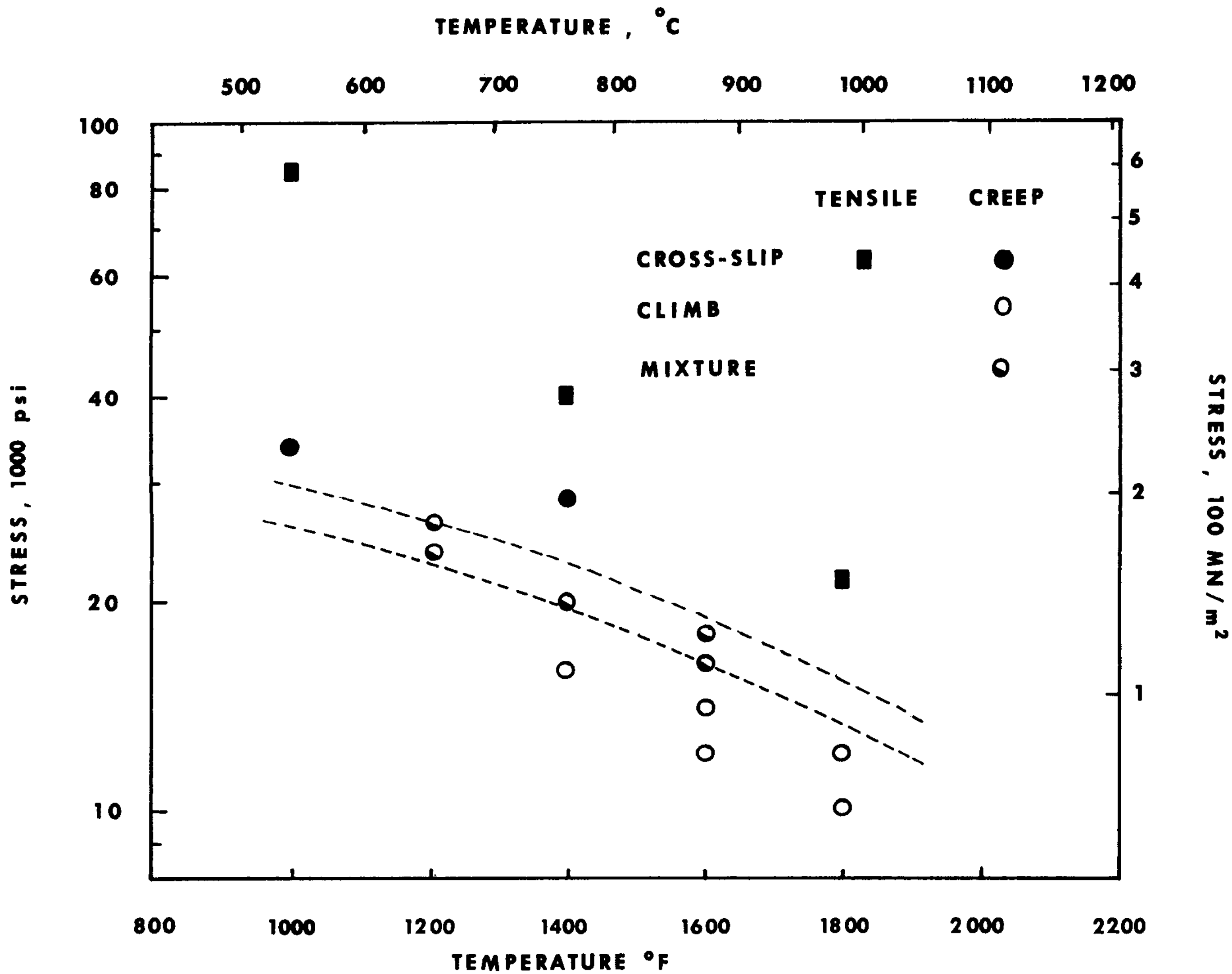
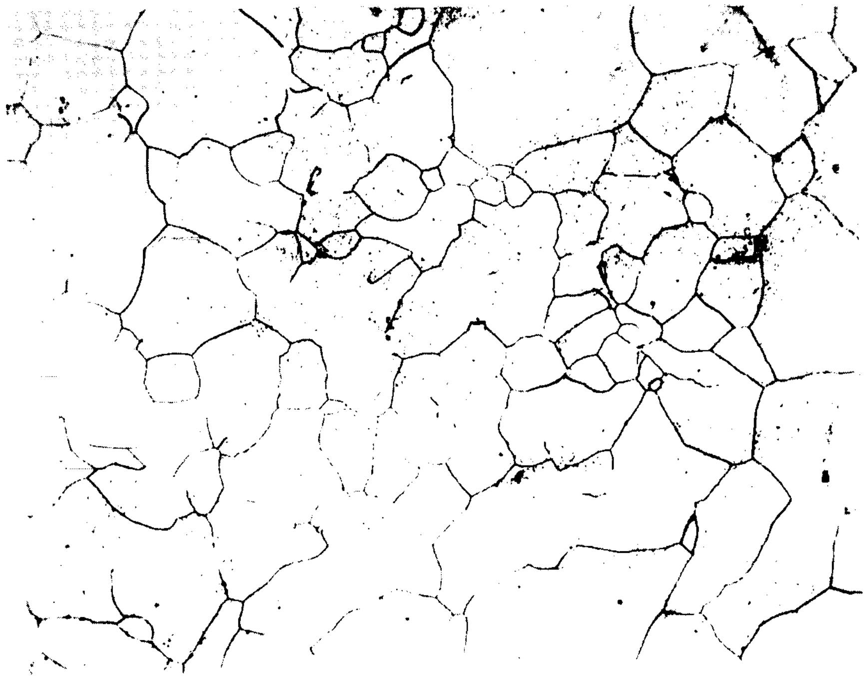
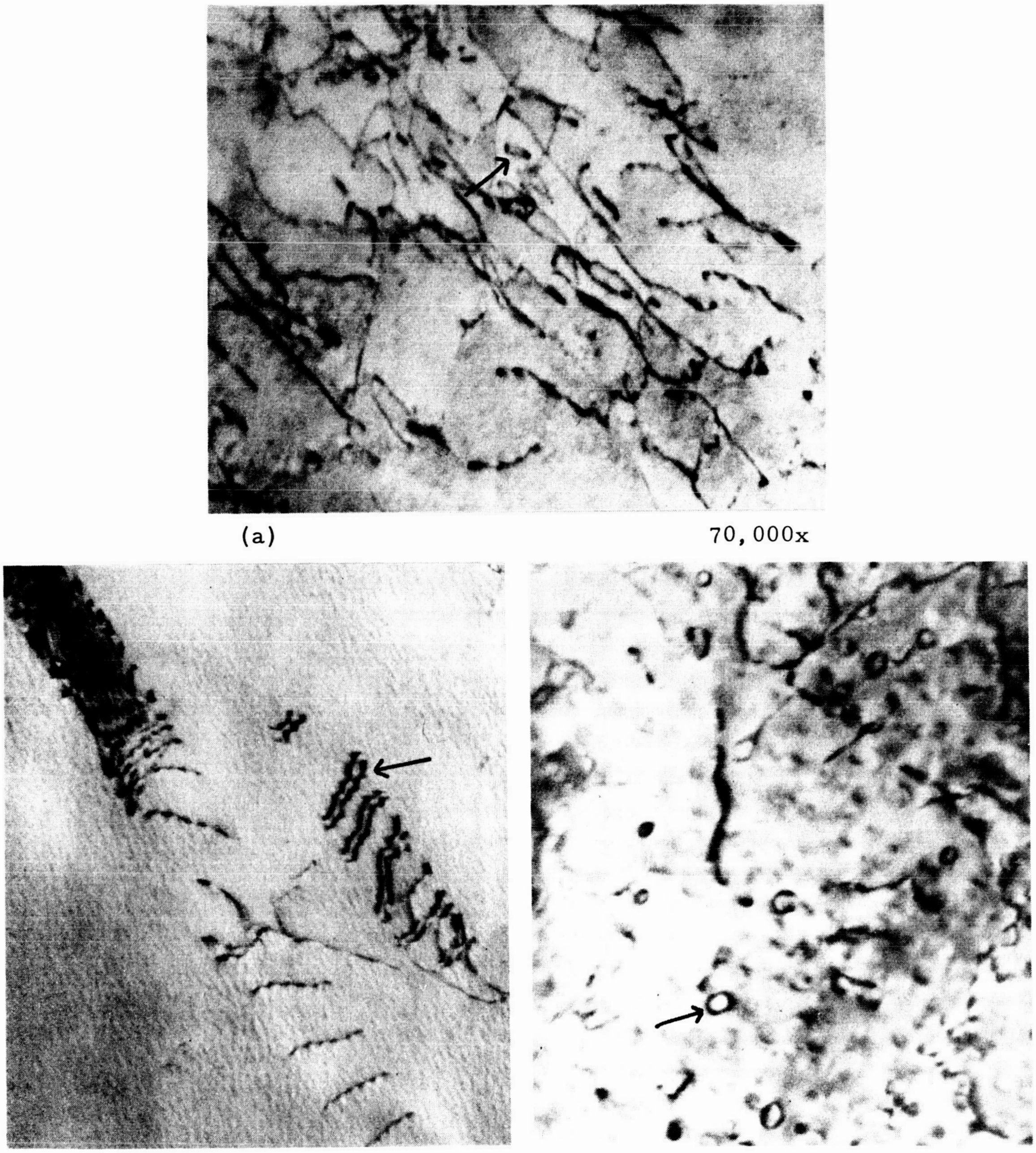


Figure 11. The variation of the dislocation mechanism observed as a function of test stress and temperature for 0.015-inch (0.38mm) thick, stress relieved, TD-Ni, Cr sheet. The mechanism changes from climb to cross-slip as the stress is increased beyond the "Orowan Stress".



250x

Figure 12. Optical photomicrograph of 0.025-inch (0.64mm) thick Modified Waspaloy sheet heat treated 1/2 hour at 1975°F (1080°C) plus 16 hours at 1400°F (760°C).



(a) 70,000x

(b) 55,000x (c) 110,000x

Figure 13. Transmission electron micrographs of thin foils from the gauge sections of smooth specimens and the shoulder of a notched specimen of Modified Waspaloy solution treated 1/2 hour at 1975°F (1080°C), aged, and creep-rupture tested. (a) smooth specimen aged 1 hour at 1400°F (760°C), tested at 90ksi. (620 MN/m^2) at 1100°F (593°C). (b)notched specimen aged 1 hour at 1400°F (760°C) tested at 60ksi. (414 MN/m^2) at 1100°F (593°C)-the stress in the shoulder section was 42ksi. (290 MN/m^2). (c)smooth specimen aged 16 hours at 1400°F (760°C), tested at 50ksi. (345 MN/m^2) at 1300° F (704°C). Prismatic dislocation loops in (a) indicate that the dislocations by-passed the γ' by cross-slip. γ' particles in (b) were sheared by super dislocations. The concentric dislocation loops in (c) were formed by dislocations bowing between γ' particles pinching off the loops.

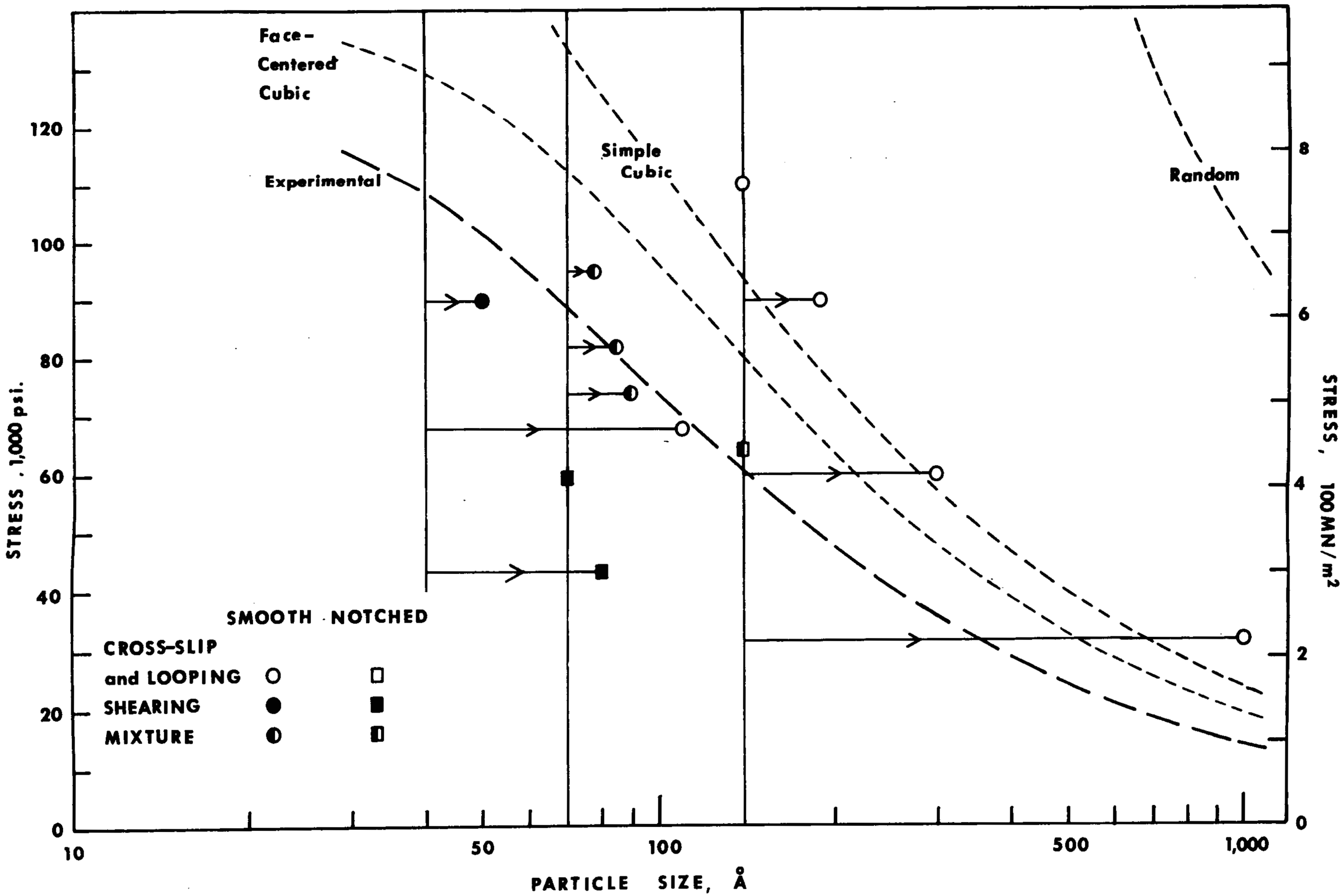


Figure 14. The variation of the dislocation mechanism as a function of test stress, compensated to 1000°F (538°C), and γ' particle size for 0.025-inch (0.64mm) thick Modified Waspaloy heat treated 1/2 hour at 1975°F (1080°C) and aged and tested at 1000° to 1400°F (538-760°C). Cross-slip and looping occurred at stresses greater than the "Orowan Stress". Theoretical values for the "Orowan Stress" varied according to the γ' particle distribution assumed.

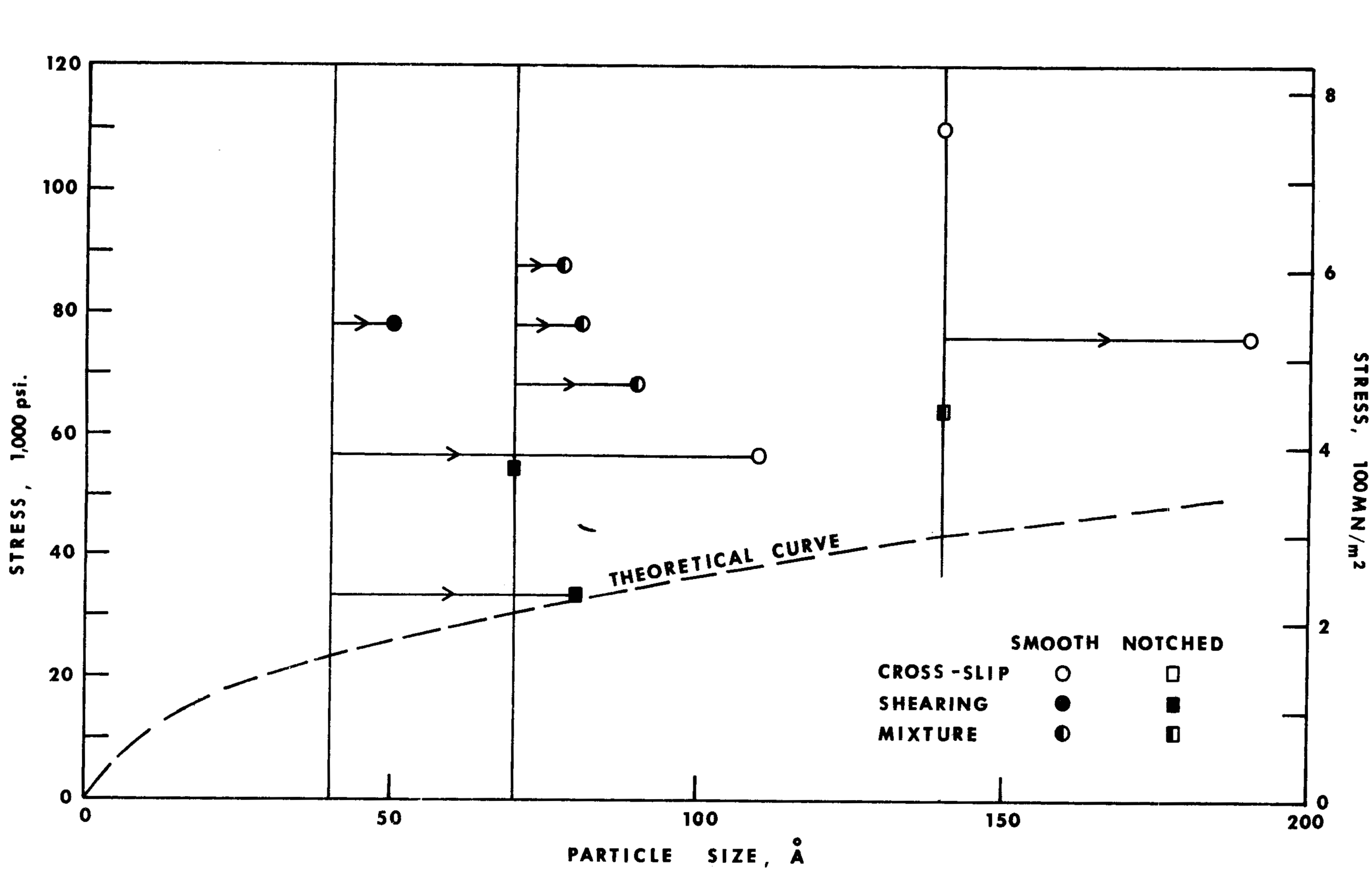


Figure 15. The variation of the dislocation mechanism as a function of test stress, compensated to 1000°F (538°C), and the γ' particle size for 0.025-inch (0.64mm) thick Modified Waspaloy heat treated 1/2 hour at 1975°F (1080°C) and aged and tested at 1000° to 1400°F (538-760°C). Dislocations can shear γ' particles when the stress is greater than a "Critical Stress".

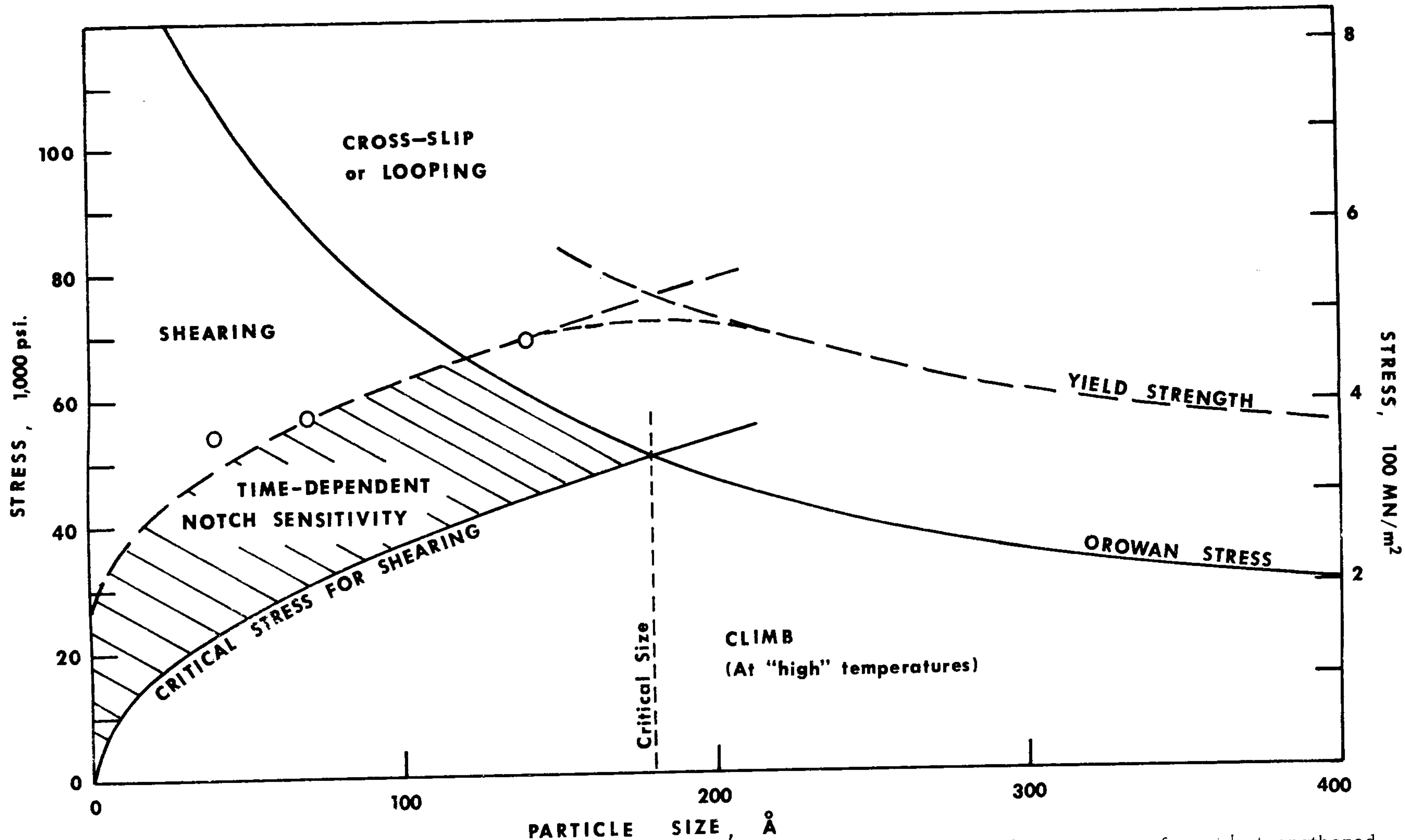


Figure 16. The dislocation mechanisms, as a function of test stress and particle size, that can occur for a γ' strengthened alloy. The curves shown are for Modified Waspaloy at 1000°F (538°C). Dislocations can shear γ' particles smaller than a critical size. Time-dependent edge-notch sensitivity has only been observed when the test stress is below the yield stress and when dislocations shear γ' particles.

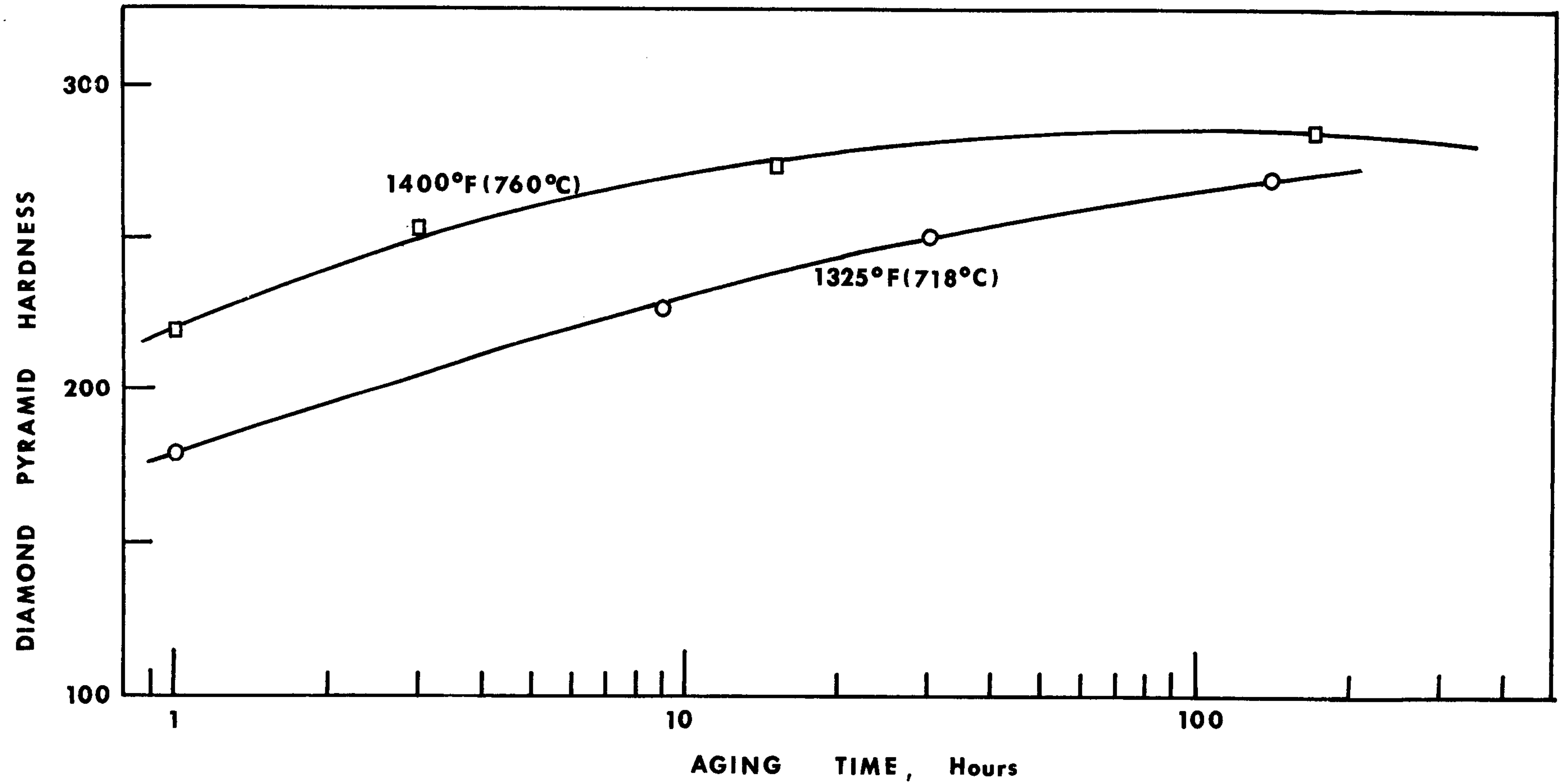


Figure 17. The effect of aging exposures at 1325° and 1400°F (718, 760°F) on the Diamond Pyramid Hardness of Modified Waspaloy solution treated 1/2 hour at 1975°F (1080°C). All of the aging treatments used in the study, 3 hours at 1325°F (718°C), 1 and 16 hours at 1400°F (760°C), resulted in γ' particles smaller than the critical size ie. maximum hardness.